Effect of Isothermal Annealing Temperature and Cooling Rate on Mechanical Properties of Ductile Iron

A Thesis Submitted in Partial Fulfillment

of the Requirement for the Degree of

Bachelor of Technology

In

Metallurgical and Materials Engineering

By

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May, 2013

National Institute of Technology

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Certificate

This is to certify that the thesis entitled "Effect of Isothermal Annealing Temperature and Cooling Rate on Mechanical Properties of Ductile Iron" being submitted by **Atul Ranjan** (109mm0125) and **Manindra Ranjan** (109mm0556) for the partial fulfillment of the requirements of Bachelor of Technology degree in Metallurgical and Materials engineering is a bona fide thesis work done by them under my supervision during the academic year 2012-2013, in the Department of Metallurgical and Materials Engineering, National Institute of Technology Rourkela, India.

The results presented in this thesis have not been submitted elsewhere for the award of any other degree or diploma.

Date: (Dr. Sudipta Sen)

Metallurgical and Materials Engineering

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CONTENTS

LIST OF FIGURES

LIST OF TABLES

ABSTRACT

A block of ductile Iron of known composition was procured and samples for various testing were cut out from that block. Tensile test specimens were machined from the block according to the ASTM E8 (Flat sub-size specimen) standard. From a set of nine specimens, three were kept untreated whereas the other six specimens were annealed isothermally following different cooling rates. All the six specimens were heated isothermally to 1000°C and held there for 90 minutes. Out of the six, three specimens were first cooled to 700°C immediately after 90 minutes and held there for 330 minutes and followed by cooling to room temperature. The rest three were immediately cooled after 90 minutes to room temperature. All the six specimens were cooled inside the furnace. Tensile test was performed at room temperature using INSTRON 1195 UTM at a crosshead speed of 1mm/min. After completion of tensile test the broken specimens were cut down to small pieces to study the fracture surface. Hardness values were recorded from Vickers hardness tester by application of 20Kg load at room temperature. X-ray diffraction technique was employed for phase analysis with Philips PANalytica x-ray diffractometer. Graphs were plotted comparing yield strength, elongation and hardness values for the "as-cast" and both the annealed samples.

Keywords: Ductile Iron, tensile testing, X-ray diffraction, SEM.

INTRODUCTION

The term "cast iron" refers to a family of materials whose major constituent is iron, with significant amounts of carbon and silicon. The carbon content of the cast iron ranges between 2 and 6.67%.Cast iron does not refer to a single material. Cast irons are natural materials whose properties are decided by their microstructure - the stable and meta-stable phases formed during heat treatment or solidification. Due to high carbon content it is very brittle in nature. By control of the alloying addition, appropriate heat treatment and good foundry practice the properties vary over a wide range. [1] The major microstructural constituents of cast irons are the morphological and chemical forms of the carbon.

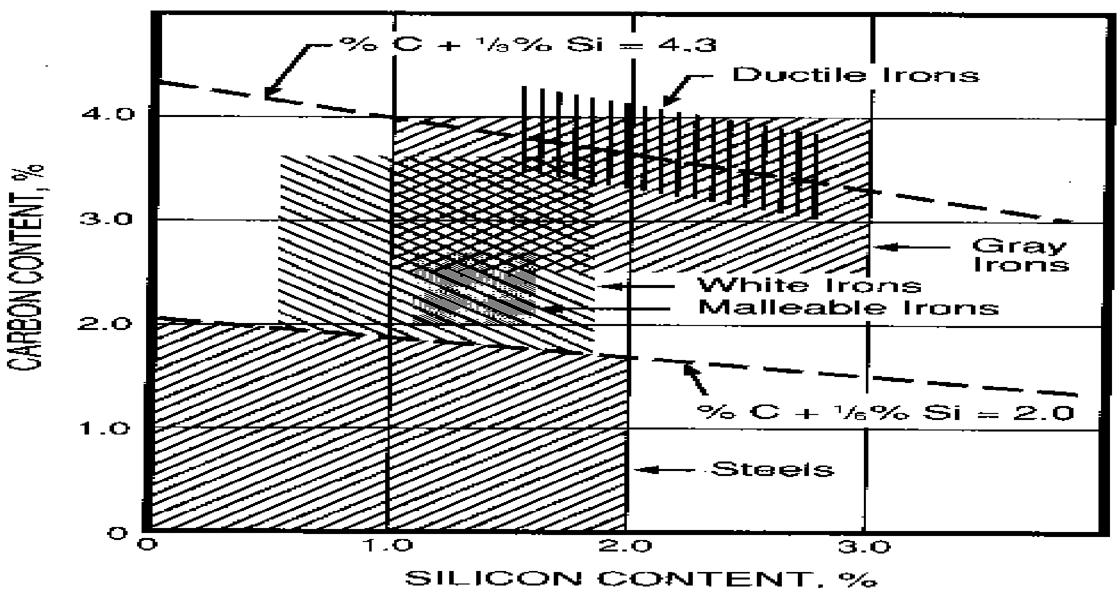


Fig 1: Variation of silicon content and type of cast iron

1.1 MICROSTRUCTURAL COMPONENTS

The following are a few of the microstructural components found in cast iron-

1.1.1 Graphite

In cast iron it is the stable form of pure carbon. Its important physical properties are low hardness, high thermal conductivity, low density, and and lubricity. The shape of Graphite present in the cast iron which may be flake or spherical, plays a significant role in deciding the mechanical properties of cast irons. Graphite flakes function as cracks in the iron matrix, whereas graphite spheroids act as "crack arresters", giving the respective irons very much different mechanical properties.

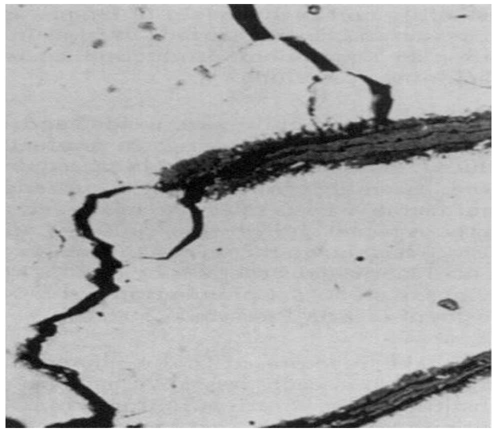


Fig2: Graphite flakes acting as crack

1.1.2 Ferrite

Ferrite is the purest form of iron in a cast iron. Generally, ductile Iron ferrite produces high ductility and toughness but lower strength and hardness. In Austempered Ductile Iron (ADI), an exceptional combination of high strength with good ductility and toughness is present because of extremely fine-grained acicular ferrite.

1.1.3 Carbide

Carbide, or cementite, is a brittle compound of carbon which is extremely hard. Strong carbide forming elements, such as molybdenum, chromium or vanadium are present. Dispersed carbides which are either lamellar or in spherical forms play a major role in deciding strength and wear resistance in as-cast pearlitic and heat-treated irons. The wear resistance of cast iron increases due to massive carbides, but this makes it brittle and very difficult to be machined.

1.1.4 Austenite

In austenitic irons, the austenitic matrix gives ductility and toughness at all temperatures, good high temperature properties and corrosion resistance especially under conditions of thermal cycling. in volume fractions up to 40% in lower strength grades stabilized austenite, improves toughness and ductility and response to surface treatments such as fillet rolling. It can exist at room temperature in austenitic and austempered cast irons, normally as a high temperature phase consisting of carbon dissolved in iron. Nickel in the range 18-36% stabilises austenite in austenitic irons. In austempered irons, a combination of rapid cooling which subdues the formation of pearlite and the supersaturation of carbon in the process of austempering, which results in depression of the start of the austenite-to-martensite transformation far below room temperature produces austenite. In austenitic irons, the austenite matrix provides ductility and toughness at all temperatures, corrosion resistance and good high temperature proper-ties, especially under thermal cycling conditions. In austempered Ductile Iron stabilized austenite, in volume fractions up to 40% in lower strength grades, improves toughness and ductility and response to surface treatments such as fillet rolling.

Apart from these phases, Bainite and Pearlite are also present in the cast irons.

1.2 Types of Cast Irons

Different types of cast iron are found depending upon the modification of solidification behaviour, presence of trace elements, heat treatment after solidification and the addition of alloying elements. These parameters change the microstructure of cast iron to produce the desired mechanical properties. Following are the common types of cast iron –

1.2.1 White Iron

White Iron is fully carbidic in its final form. The presence of different carbides, produced by alloying, makes White Iron extremely hard and abrasion resistant but very brittle.[2]

1.2.2 Malleable Iron

Malleable Iron is cast as white iron and an annealing or "malleablizing" heat treatment is required to convert the carbide into graphite. This is in contrast with grey cast iron. The microstructure of Malleable Iron consists of irregularly shaped nodules of graphite called "temper carbon". Causes of its reduced casting yield and increased production costs are the formation of carbide during solidification which results in the shrinkage behaviour.

1.2.3 Gray Iron

Gray Iron, named because its fracture has a gray appearance, consists of carbon in the form of graphite flakes in a matrix consisting of ferrite, pearlite or a mixture of these two. Gray Iron is by far the oldest and most common form of cast iron. The flakes' shape of graphite in Gray Iron exerts a large influence on its mechanical properties. Gray Iron exhibits no elastic behaviour and fails in tension without significant plastic deformation. This is because graphite flakes can act as stress raisers which may cause localized plastic flow at low stresses prematurely. Gray Iron has good machinability, damping and self-lubrication properties.

1.2.4 Ductile iron

It is also known as ductile cast iron, nodular cast iron, spheroidal graphite iron, spherulitic graphite cast iron and SG iron The most important and remarkable microstructural feature of all Ductile Irons is the presence of graphite nodules which act as "crack-arresters" and give Ductile Iron ductility and toughness superior to all other cast irons, and The nodules are formed during solidification, not during heat treatment. The type of matrix in which the graphite nodules are dispersed plays an important role in determining mechanical properties of Ductile Iron.

1.3 History of Ductile Iron Development

The search for an ideal cast iron - an as-cast "gray iron" with mechanical properties equal or superior to Malleable Iron led to the discovery of Ductile Iron. Conventionally, a ladle of magnesium is added (as an alloy of copper-magnesium) to cast iron causing the solidified cast pieces that do not comprise of graphite flakes, but almost spheres of graphite. Hence, Ductile/S.G Iron was discovered . The discovery took place in the year 1943 in International Nickel Co. Research Lab by Keith millis.[3]

1.4 Advantages of Ductile Iron

There are many advantages of Ductile/S.G Iron which contribute to its success. Some of these advantages are- versatility and higher performance at lower cost, high ductility and strength. Other varieties of the ferrous cast iron category may have individual properties which make them the best material in some applications, but the versatility of Ductile/S.G Iron is much more. It provides the best combination of all round properties. It can be made to have high ductility, with some grades assuring more than 20% high strength or elongation, with tensile strength in excess of 830 MPa. Austempered Ductile Iron (ADI), does provide tensile strength above 1600 MPa. It offers superior mechanical properties and good wear resistance.

1.5 Types of Ductile Iron

Ductile/S.G Iron is a collection of materials having a broad range of properties gained by microstructural control. The features common to all types of these are the almost spherical graphite nodules shape. Ductile Iron matrix determines mechanical properties - when a good amount of graphite nodules present in the material.[4] The utilisation of matrix names to categorise the types of Ductile/S.G Iron shows the important role of the matrix in deciding mechanical properties.

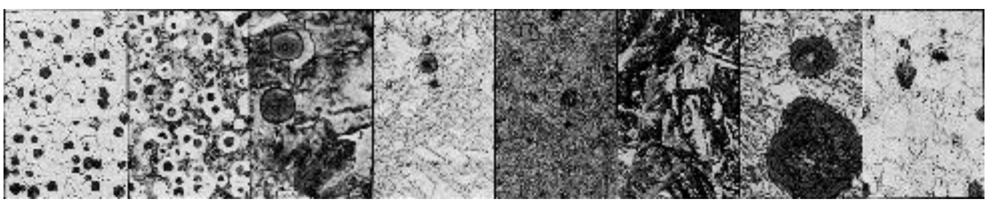


Fig 3: Microstructures of various types of Ductile Iron.

1.5.1 Ferritic Ductile Iron

In Ferritic Ductile/S.G Iron ,graphite spheroids which are present in a matrix of ferrite causes an iron to have good impact resistance and ductility with the value of yield strength and tensile strength almost being equal to a low carbon grade steel. It may be manufactured "as-cast" but can also be given an annealing treatment to ensure the max. ductility and toughness at low temperatures.

1.5.2 Ferritic Pearlitic Ductile Iron

These are the most general grade of Ductile/S.G Iron, normally being produced in "as cast" conditions. The spheroids of graphite are present in a pearlite and ferrite matrix . Properties shown are in between the ferritic and the pearlitic grades of iron , these have low production costs and good machinability.

1.5.3 Pearlitic Ductile Iron

These are iron with high strength, superior wear resistance, and moderate impact resistance.[5] Spheroids of graphite are present in matrix made up of pearlite in this type of Iron. Greater machinability than steels of approx. same properties.

1.6 PRODUCTION OF DUCTILE IRON

Molten alloy is treated with cerium, or magnesium or their combination which causes spheroidal graphite growth during solidification. Ba, Li, Ca, Zr may also be used for this purpose. if elements, like Ti, 0.009% Pb, Bi or 0.004% Sb are present then they prevent the production of ductile iron , but addition of 0.005-0.01% Ce removes their effect. Adding of ferro-silicon is done after about 0.06% residual content is obtained through addition of Mg. combination of magnesium and cerium may also be used and after that ferro silicon may be added so as to produce ductile iron.

1.6 .1 Desulfurisation

Graphite flakes formation is caused by Sulphur. So the raw material should have low sulfur percentage (<0.10 %). Sulphur removal may be done during melting or by adding a desulphurising agent like soda ash ($NaNO_3$) or calcium carbide.

1.6.2. Nodulising

To remove sulfur and oxygen which is still present in liquid alloy Magnesium is added and it provides a residual 0.05% of magnesium, this results in growth of spheroidal graphite , probably the interfacial energy has a high value to have a 180 degree this implies that the wetting of graphite does not occur.This treatment with magnesium desulphurises iron to less than 0.02% . Magnesium and other similar elements have a appreciable affinity for sulfur , and so the scavenge sulfur from the molten alloy as an initial stage for producing Ductile Iron.

1.6 .3. Inoculation

Being a carbide former, in the form of magnesium being present ,ferrosilicon is immediately added as an innoculant. Reversion to graphite flake due to loss of magnesium is caused by Remelting. Molten alloy stirring after nodularising element is added evolves large amount of gas, which can get dissolved in liquid alloy, and solid casting blow-holes are formed.

1.7 Properties of Ductile Iron-

Tensile Property

Generally, tensile properties of Ductile/S.G Iron - elongation and the tensile and yield strengths, are mostly referred parameters regarding mechanical behaviour. Many of the specifications world-wide for Ductile/ S.G iron illustrate the properties of the various different grades of Ductile/S.G Iron by their respective tensile strength and yield strength values and also the elongation behaviour. The values of hardness, is also offered, but that is specified only for some ferritic grades is the impact properties. Other parameters like the proportional limit and modulus of elasticity are also considered as vital criteria for designing purposes. [7]

1.7.1 Modulus of Elasticity

, A proportional or linear relationship , At low value of tensile stresses ,is observed between stress and strain. This relationship is called the Hooke's Law and the value of slope of the straight line obtained is called the Young's Modulus or Modulus of Elasticity . [8]The stress-strain behaviour, in initial stages, of Ductile /S.G Iron lies between that of Grey Iron and mild steel. Normalized or annealed mild steels show elasticity in behaviour up till the yield point, whereas the plastic deformation happens suddenly and without showing signs of any initial increment in flow stress.

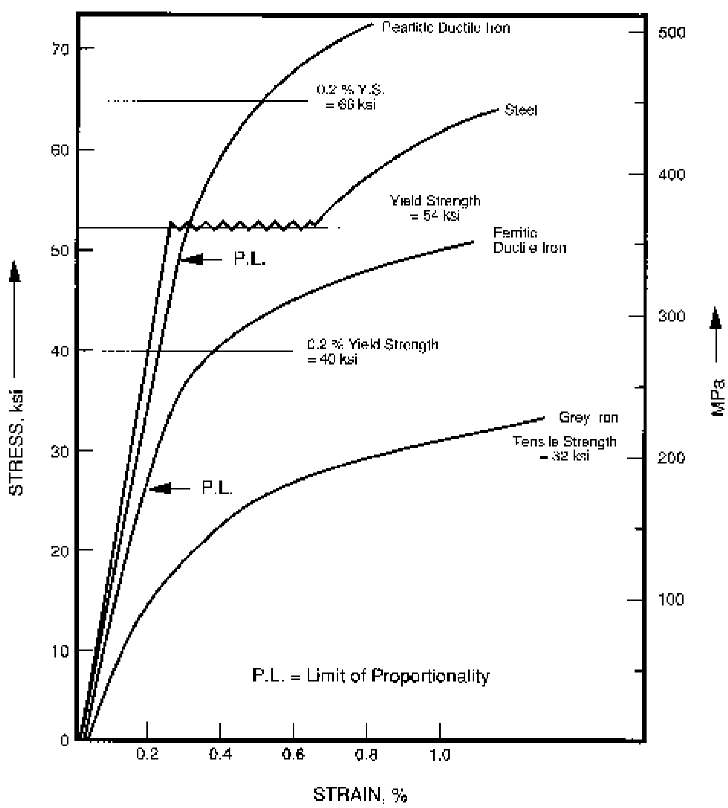


Fig 4: Elastic and yielding behavior for steel, Gray Iron and ferritic and pearlitic Ductile Irons

1.7.2 Poisson's Ratio

Poisson Ratio, defined as the ratio between lateral elastic strain and longitudinal elastic strain shown in a tensile test, shows very little fluctuation in the case of Ductile Iron. A common value accepted is 0.275.

1.7.3 <u>Proportional Limit</u>

Proportional limit or the” limit of proportionality” is the maximum value of stress at which any material shows elastic behaviour. If the material is stressed below the proportionality limit, and then the stress is removed, the stress-strain curve retraces back to the origin – showing that no permanent dimension change occurring. When the applied stress is above the proportionality limit, the slope of the stress-strain curve is reduced due to the plastic strain. On removal of the applied stress, the strain value comes down linearly, tracing a line parallel to the earlier shown elastic curve. At zero applied stress, the strain does not reduces to zero, thus showing a permanent plastic strain, or a permanent change in the specimen dimension.

1.7.4 <u>Yield Strength</u>

The yield strength value or the proof stress is the value of stress at which any material begins to show significant plastic deformation. The rapid transition from elastic behaviour to plastic behaviour shown by annealed as well as normalized steels helps to determine yield strength almost accurately. For Ductile/S.G Iron the method used is the offset one in which the measurement of yield strength is at a pre-determined deviation from the original linear relationship shown between stress and strain. This deviation, generally taken as 0.2 %, is also included while defining the yield strength in international specifications and is also often included in the terminology for yield strength, "0.2 % yield strength". For Ductile Iron, yield strengths may range from 275 MPa for ferritic grades to over 620 MPa for martensitic grades of iron.

1.7.5 <u>Tensile Strength</u>

The tensile strength also called ultimate tensile strength (UTS), is the value of maximum amount of load which a material can withstand in tension, prior to fracture. It is determined by dividing the maximum value of load applied during the tensile testing by the initial area of the cross section of the sample. Tensile strengths for Ductile Irons generally range 414 MPa for ferritic grades to above 1380 MPa for martensitic grades of iron.

1.7.6 <u>Elongation</u>

Elongation is the permanent increment in length, expressed as the percentage of gage length specified, which is marked in a tensile testing bar, which is shown in failure testing of the bar. Elongation is used as an indication of tensile ductility and is also included in many specifications of Ductile Iron. It also includes the localized deformation occurring prior to the fracture. Since the localized deformation happens in a limited part of gage length, its contribution to the total value of elongation of a test bar is very small. Brittle materials such as Gray Iron may fail in tension without showing any significant elongation, but ferritic Ductile/S.G Irons can show elongation of about 25 %. [9]

1.8 EFFECT OF VARIOUS PARAMETERS ON PROPERTIES OF DUCTILE IRON

1.8.1 Effect of Graphite Shape

As would be expected from the dramatic differences in mechanical properties between Gray and Ductile Irons, that modularity plays a significant role in determining properties within the Ductile Iron family. There correlation between modularity and Dynamic Elastic Modulus.[6] This relationship not only emphasizes the strong influence of modularity on DEM, but also indicates that DEM values obtained by sonic testing can be used to measure modularity (graphite volume and nodule count should be relatively constant).

Nodularity, and the morphology of the non-spherical particles produced as modularity decreases, exert a strong influence on the yield and tensile strengths of Ductile Iron modularity has been changed by two methods: through magnesium control, or through lead control. When nodularity is decreased by agent used in commercial Ductile Iron) the nodules elongate and do not become sharp or "spiky". This is a 10% decrease in yield strength as well as a 15% decrease in tensile strength where modularity is reduced to 30%. Meagre additions of lead lessen modularity by creating intergranular networks of "spiky" or plate-like graphite which result in dramatic decrease in tensile properties.

1.8.2 Effect of Nodule Count

Nodule Count, defined mathematically as the number of graphite nodules/MM^2, also influences the mechanical properties of Ductile Iron but not as strongly and directly as graphite shape. In general, high nodule count shows good metallurgical quality, albeit there is an optimum range of nodule count for every casting, and large proportion of it may result in a degradation of properties. Nodule count per se mildly affect tensile properties, although it affects the microstructure, which can largely influence properties.

- Nodule count has inverse relationship with the pearlite content of as-cast Ductile Iron. Increasing the nodule count decreases the pearlite content, depreciating strength and enlarging elongation.
- Nodule count also affects carbide content. Increasing the nodule count enhances tensile strength, ductility and machinability by decreasing the volume fractions of chill carbides, segregated carbides, and carbides related to "inverse chill".

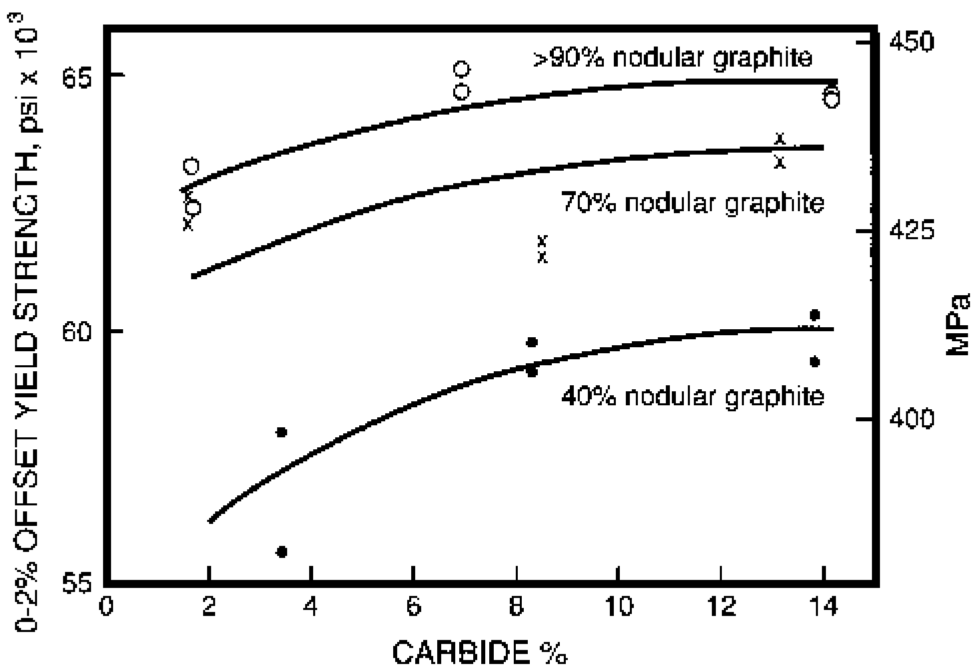


Fig 5: Relationship between tensile strength and hardness of Ductile Iron. Wu indicates percent of sample exceeding indicated tensile strength for a constant hardness.

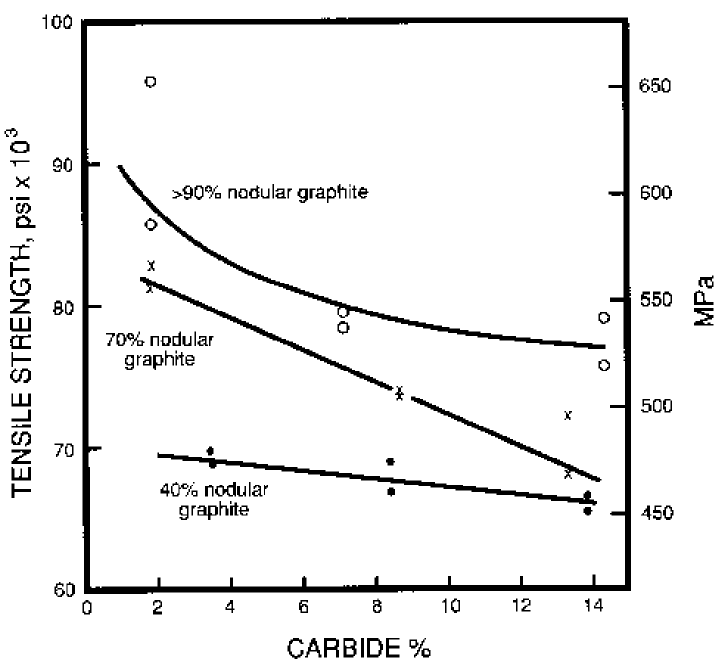


Fig 6: Effect of nodularity and carbide content on tensile strength of pearlitic Ductile Iron.

1.8.3 <u>Effect of Graphite Volume</u>

Volume fraction of graphite in S.G Iron affects some of the tensile properties. Increasing the carbon content enlarges the volume fraction of graphite, reducing the DEM for a fixed section size. Casting section size alter both the volume fraction as well as size of graphite nodules. Enhanced section size decreases the cooling rate of the cast, causing carbon to precipitate in the stable graphite phase and not the carbide phase increased by higher cooling rates. The reduced cooling rates of the increased diameter bars too affect graphite nucleating environment, producing reduced nodule count but enhanced nodule size. The increment in nodule size at par with section size is the chief reason for the reduced DEM, but enhancement in the formation of graphitic carbon during solidification would also add to it.

1.8.4 <u>Effect of Carbide Content</u>

Carbide content affects the properties directly or indirectly. Enhancing the volume fraction of hard, brittle carbide enhances the yield strength, but lessens the tensile strength of gray iron castings. Carbides available in a Ductile Iron matrix too enhance the dynamic elastic modulus and decrease machinability by large. The build up of eutectic carbide during solidification has an effect on the volume fraction of graphite generated as carbide and graphite vie for the carbon present in the liquid iron.

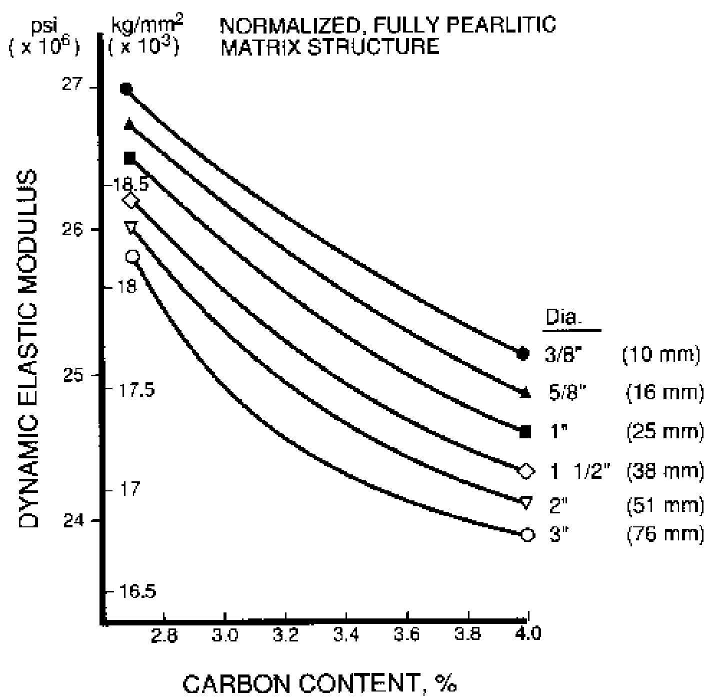


Fig 7: Effect of carbon content and casting diameter on the dynamic elastic modulus of fully pearlitic Ductile Iron.

1.8.5 Effect of Matrix

Ductile Irons which have fixed modularity, nodule count, low porosity and carbide content, mechanical properties are calculated chiefly by the matrix contents and their hardness. General grades of Ductile Iron, the matrix contain ferrite and/or pearlite. Purest iron phase in Ductile Iron is that of ferrite. It shows low strength and hardness, but high ductility and toughness and appreciable machinability. Pearlite is a thorough mixture of lamellar cementite in a matrix of ferrite. When compared with ferrite, pearlite shows higher strength and hardness and lower ductility. The mechanical properties of ferritic/pearlitic Ductile Irons are, thus, determined by the ratio of ferrite to pearlite in the matrix. [10]This ratio is checked in the as-cast condition by checking the composition of the iron, taking into account the cooling rate of the casting. It may well be handled by an annealing heat treatment which generates a solely ferritic casting, or by normalizing to increase the pearlite content.

1.8.6 Effect of Temperature

Deciding design stresses for a Ductile Iron section, the designer must have the knowledge of the temperature range under which the component will be functioned and the outcome of temperature on tensile behavior. Enhancement in yield strength with reducing temperature for both ferritic and pearlitic, Ductile Irons recommends that complex design stresses be brought into effect at low temperatures. As maximum low temperature applications also comprise of performance at room temperatures, the room temperature yield strength should be utilized in the determining design stresses. Although, utilization of a yield strength-related design stress is suitable for low temperature applications only when the applied stress state can be simulated by a quasi-static (low strain rate) test.

1.9 PHYSICAL PROPERTIES

1.9 .1 Density

The normally accepted value of density at the room temperature of S.G Iron is 7.10 gm/cc. Density is influenced manly by the amount of carbon which is graphitized, with fluctuating densities from 7.40 gm/cc for high carbon ferritic and 6.80 gm/cc for low carbon pearlitic irons. The density of a cast steel which is 7.8 gm/cc - is higher than that of S.G Iron by almost 10 per cent. A steel casting or forging can be replaced with a lighter S.G Iron casting increasing the strength: weight ratio of the component, also, energy savings and costs throughout lifetime, especially in components such as crankshafts in an automotive.

1.9.2 Thermal Expansion

The linear thermal expansion coefficient of S.G Iron depends basically on its microstructure, also, it is influenced to a lesser extent by structure of graphite and temperature too. In unalloyed S.G Iron, volumetric composition has only a little effect on thermal expansion, but alloyed austenitic S.G Irons exhibit appreciably different behaviour of expansion.

1.9.3 Thermal Conductivity

The thermal conductivity of Ductile Irons are affected strongly by morphology of graphite. Same is the case with electrical properties. [11]The conductivity value is slightly higher in Grey Iron because of the discontinuous nature of flakes of graphite. Owing to the influence of graphite flakes on the conductivity, the graphite volume fraction of the material plays a significant role in Grey Iron, but not in S.G Iron. Other than the shape of graphite, its microstructure, temperature and composition also impact the value of thermal conductivity. Ferritic S.G Irons have a greater thermal conductivity when compared to pearlite grades, and tempered and quenched irons have values between those of pearlitic and ferritic irons. In the range 20-500 degree Celsius, the thermal conductivity of ferrite grades is 35 W/molK.

1.9.4 Specific Heat

Specific heat, defined as the amount of energy necessary to enhance the temperature of unit mass of a material by 1 degree, normally increases with temp. , reaching a maximum point when a phase transformation takes place.[12]

1.9.5 Electrical Resistivity

S.G Irons, having discontinuous spheroidal graphite, show low electrical resistivity as compared to Grey Irons which possess semi-continuous type flake graphite structure. The main elements influencing resistivity are nickel and silicon both of which enhance resistivity.

1.9.6 Magnetic Properties

The properties are ascertained mainly by the microstructures in case of S.G Irons. The shape of graphite particles i.e spheroidal in S.G Irons gives them higher permeability and higher induction both, in comparison to Grey Irons which have a similar matrix. For greater permeability and reduced loss of hysteresis, low phosphorus ferritic irons are used. Ferritic S.G Irons are softer than pearlitic grades magnetically, i.e, they have low hysteresis loss and higher permeability.

1.9.7 Wear Resistance

Mechanical wear is the surface deterioration and material loss due to stresses generated from surfaces of two bodies in contact. Wear is basically mechanical in nature but it may also involve chemical reactions.

Cast irons are regarded since many years as an ideal material for diverse wear applications, under both dry and wet conditions especially frictional wear. In dry wear, appreciable amount of heat may be generated due to the friction between the contact surfaces to harden the surfaces or, in some cases, fuse them together. Under these environment the graphite present in cast irons lubricate the interface , reducing frictional force and also the surface deterioration due to overheating.

1.9.8 Corrosion Resistance

Unalloyed S.G iron shows approx. the same resistance to corrosion as Grey Iron and are better over unalloyed steel, and highly alloyed steel in certain conditions. Corrosive atmosphere degrades the performance of S.G Iron in several ways: the embrittlement of stressed components and the loss of structural and material integrity caused only by corrosion. Corrosion also has a major role in abrasive wear resistance of the material.

1.10 HEAT TREATMENT

Heat treatment is a valuable and versatile tool for extending both the consistency and range of properties of Ductile Iron castings beyond the limits of those produced in the as-cast condition. the graphite spheroids in Ductile Iron play a critical role in heat treatment, acting as both a source and sink for carbon. [13]When heated into the austenite temperature range, carbon readily diffuses from the spheroids to saturate the austenite matrix. On slow cooling the carbon returns to the graphite "sinks", reducing the carbon content of the austenite. This availability of excess carbon and the ability to transfer it between the matrix and the nodules makes Ductile Iron easier to heat treat and increases the range of properties that can be obtained by heat treatment.

1.10.1 Austenitizing

It is the heat treatment procedure where the ferrous alloy is held above the upper critical temperature for a adequately long time to ensure that the matrix has wholly transformed to austenite. It is carried out before a heat treatment process to produce a uniform homogeneous matrix.

1.10.2 Tempering

It is the heat treatment process in which the quench-hardened or normalized ferrous alloy is reheated to a temperature below the transformation temperature and cooling is done at any preferred rate. Tempering is done to relieve thermal residual stresses and for enhancing ductility & toughness. The enhancement in ductility by tempering leads to reduction in the hardness & strength. These deviations in properties are attained by holding the castings at a temperature that is below the critical temperature. Tempering is a diffusional process and thus is time and temperature dependent. Tempering conditions are swayed strongly by the anticipated change in properties, the alloy content, the microstructure being tempered and the nodule count. Low alloy content, martensitic structures and high nodule count reduce tempering temperatures and/or times, though high alloy content, a normalized (pearlitic) structure and low nodule count intensify tempering times.

1.10.3 Normalizing

Normalizing (air cooling following austenitizing) can cause a significant enhancement in tensile strength and may be used in the production of ductile iron. The microstructure attained by normalizing is determined by on the composition of the castings and the cooling rate. Normalizing generally produces a homogeneous structure of fine pearlite, if the iron is not too high in silicon content and has at least a modest manganese content (0.3 to 0.5% or higher). Heavier castings that necessitate normalizing typically comprise of alloying elements such as nickel, molybdenum, and further manganese, for greater hardenability .Lighter castings made of alloyed iron may be martensitic or may have an acicular structure after normalizing.[14]

The normalizing temperature is generally between 870 and 940°C (1600 and 1725°F). The standard time at temperature of 1 h per inch of section thickness or 1 h minimum is usually satisfactory. Longer times may be required for alloys containing elements that retard carbon diffusion in the austenite.

1.10.4 <u>Annealing</u>

Annealing softens Ductile Iron by producing a carbide-free, fully ferritic matrix. These procedures range from a low temperature or sub-critical anneal used to ferritize carbide-free castings, to two-stage and high temperature anneals designed to break down carbides. The primary purpose of annealing, or ferritizing, [15]Ductile Iron is the production of castings with maximum ductility and toughness, reduced strength and hardness, improved machinability and uniform properties. Annealing castings with different levels of copper and tin has reduced strength and hardness, increased elongation, and generally eliminated the variations in as-cast properties produced by the different alloy levels.

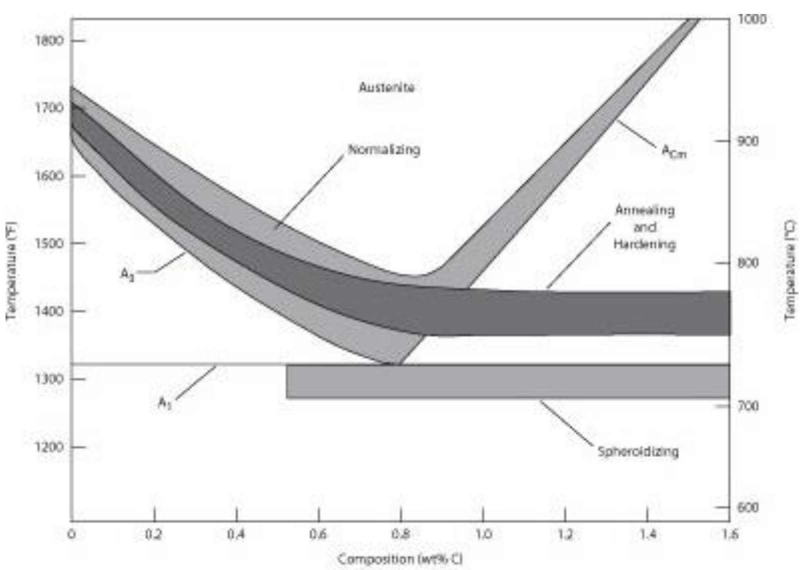


Fig 8: Steel heat treating ranges

LITERATURE SURVEY

Much of the work has been done on sg iron and enhancement of its properties by the heat treatment. Surveys of various literature shows various tempering temperature and austempering methods employed to bring about the increase in ductility , strength and toughness values.

Austenitising temp: 850°C Austenitising time: 1 hr		Hardness Values(RA)	
Austenitising temp(°C)	Austenitising time(hr)	Sample 1	Sample 2
250	0.5	56	63
	1.0	64	73
	1.5	62	71
300	0.5	54	59
	1.0	63	39
	1.5	61	78
350	0.5	53	56
	1.0	61	67
	1.5	59	65
400	0.5	46	54
	1.0	56	63
	1.5	53	60

Table 1: Hardness values for different austenising temperatures and austenising time

From the table obtained by plotting the hardness values vs. the tempering temperature that the hardness value of the specimen tempered it is clear that more is the tempering temperature, less is the hardness or more is the softness (ductility) induced in the quenched specimen. It can also be observed that keeping the tempering temperature same (either 250 degree or 300 degree or 350 degree or 400 degree), the hardness value decreases with the increase in tempering time.
It can be inferred that more is the tempering time (keeping the tempering temperature constant), more is the ductility induced in the specimen.Infact from the above two inferences it is quite clear that ductility increases with the increase in tempering temperature and also tempering time.
It can be observed that For smaller austempering times, during the initial stage, stage I reaction proceeds and the amount of bainitic ferrite and high carbon austenite gradually increases. But carbon enrichment in retained austenite is too less to make all the retained austenite stable at room temperature and some transformation to martensite is involved. With the increase in austempering time, the amount of retained austenite and bainitic ferrite increases untill completion of bainitic transformation resulting in increase in hardness, tensile strength and yield

strength. After completion of bainitic transformation, if austempering is continued for still longer duration, stage II reaction sets in and retained austenite decomposes to bainitic ferrite and carbide. This results in decrease of hardness, tensile strength and yield strength after achieving a peak value.

Thus, if hardness is the only criteria than we should go for tempering at 250 degree centigrade (tempering time 1 hour). However an optimum combination of hardness and ductility is desired, we should go for austempered specimen.

Aust Temp(°C)	Aust Time(hr)	UTS(Mpa)	Y.S(Mpa)	EL(%)
250	0.5	927	689	2.1
	1.00	1077	847	2.6
	1.50	1065	821	2.8
300	0.50	767	529	3.7
	1.00	921	686	4.3
	1.50	901	648	4.6
350	0.5	649	434	6.1
	1.00	809	561	6.9
	1.50	795	539	7.1
400	0.50	588	379	5.5
	1.00	766	521	5.8
	1.50	742	497	5.6

Table 2:Tensile properties of ADI with Tempering Temp

It can be observed from the table that for a particular tempering temperature with increase in tempering time the UTS gradually decreases and the same thing happens to the yield strength and on the other hand elongation of the specimen increases which signifies that more ductility is induced with increase in tempering time.

This clearly implies that the UTS and also to some extent the yield strength decreases with increase in tempering time where as the ductility(% elongation) increases. It can be seen that keeping the tempering time same but on increasing the tempering temperature (from 250 degree centigrade to 650 degree centigrade), the UTS value and the yield strength gradually decreases where as there is a gradual increase in % elongation.

As a result of the above observation one can clearly infer that for a given tempering time, an increase in the tempering temperature decreases the UTS value and the yield strength of the specimen where as on the other hand increasing the % elongation and hence the ductility. It is seen that as a result of the special type of heat treatment given to the austempered specimen, the yield strength of the specimen is maximum among all the quenched, and normalized specimen. The strength obtained is even more than the maximum strength obtained among all heat treated specimen i.e. tempered at 250 degree centigrade for 1 hour.

Thus it is quite clear that if an optimum combination of properties i.e. UTS, Yield Strength, Hardness and ductility (% elongation) is desired, austempering of the S.G cast iron is the one that we should go for.

Not much of work has been done in the field of annealing which includes isothermal annealing on the microstructure and mechanical properties . Therefore this project was carried out keeping in mind to obtain the effects of annealing on the microstructure of S.G iron.

EXPERIMENTAL WORK

3.1 <u>Experimental procedure</u>

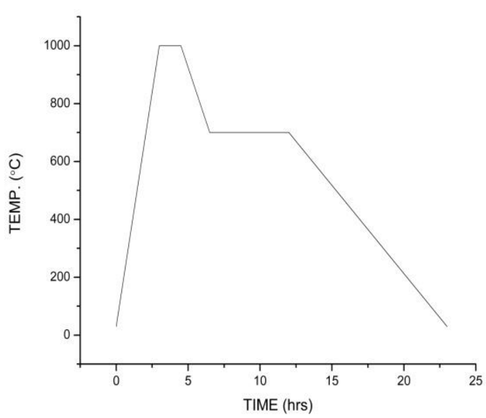
Tensile test specimens were machined from a test block according to the ASTM E8 (Flat sub-size specimen) standard. The alloy in this study consists of C-3.45%, Si-2.07%, Mn-0.15%, S-0.008%, P-0.024%, Cr-0.02%, Ni-0.15%, Mg-0.043%. From a set of nine specimens, three were kept untreated whereas the other six specimens were annealed isothermally following different cooling rates.

Element	Percentage(wt%)
C	3.45
Si	2.07
Mn	0.15
S	0.008
P	0.024
Cr	0.02
Ni	0.15
Mg	0.043

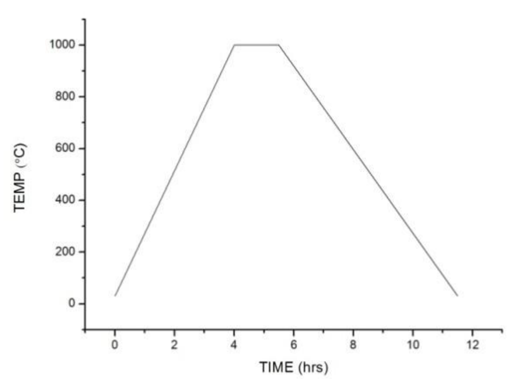
Table3: Chemical Composition used for the study

3.1.1 <u>Heat treatment</u>

All the six specimens were heated isothermally to 1000°C and hold there for 90 mins. Out of the six three specimens were first cooled to 700°C immediately after 90 mins and hold there for 330 mins. & followed by cooling to room temperature. The rest three were immediately cooled after 90 mins to room temperature. All the six specimens were cooled inside the furnace. Both heat treatment processes are shown in fig.1 & fig.2 respectively.



(a)



(b)

Fig 9: (a) – heating the specimen to 1000°C and holding there for 90 mins, cooling to 700°C, holding there for 330 mins followed by furnace cooling.
(b) – heating the specimen to 1000°C and holding there for 90 mins followed by furnace cooling.

3.1.2 Tensile test

Tensile test was performed at room temperature using INSTRON 1195 UTM at a crosshead speed of 1mm/min. Using a electronic slide caliper the thickness and the total length of the specimen was measured. The diameter of the specimen and the gauge length which was fixed at 50mm was fed to the testing machine. The distance between the jaws was fixed according to the gauge length of the specimen. The specimen was gripped by the jaws and axial load was applied to it. Ultimate tensile strength, 0.2% yield strength was obtained directly from the computer integrated to the m/c, after completion of test. Percentage elongation was calculated by dividing the displacement at break to the gauge length. The average value of three readings for each type of treated and untreated specimen is presented in table 1 along with the hardness values.

3.1.2.1 Specification of tensile testing specimen:

The tensile testing specimen as per the Indian Standard is collected.
The specification is as per the ISO 1608: 1995 ANNEX C.

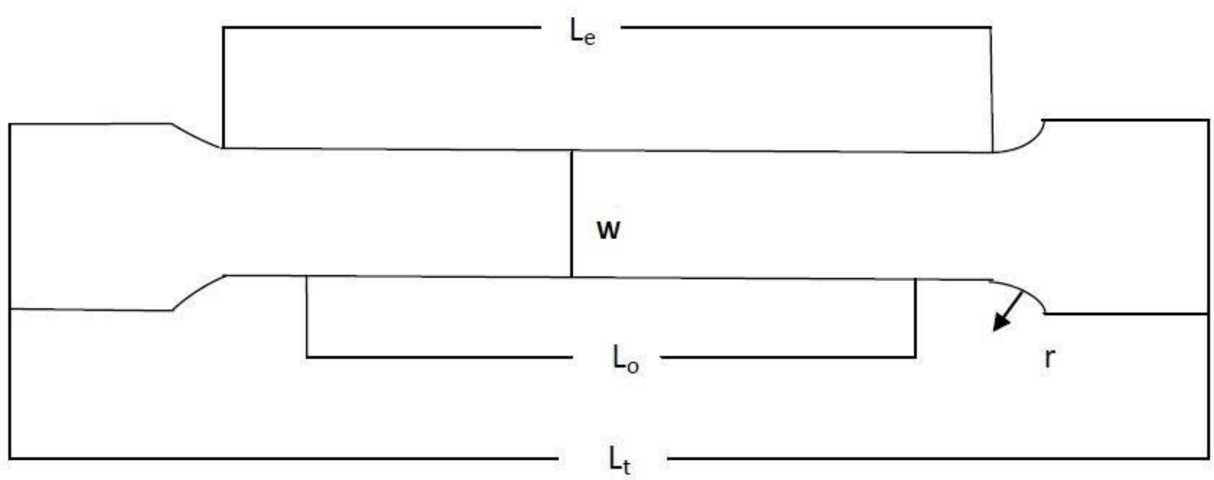


Fig 11: shape of tesile specimen

L_t = overall length =101.6 mm
w = width of the parallel section =6.35 mm
r = fillet radius =6.35 mm
Length of grip = 31.75 mm
Width of grip = 9.525 mm
L_o = gauge length = 25.4 mm

3.1.3 Fractography

After completion of tensile test the broken specimens were cut down to small pieces to study the fracture surface. The fractured area for all the three types of specimen was put under scanning electron microscope (SEM) and was analyzed at 50, 100, 250 & 500 magnification levels. The SEM micrographs are shown.

3.1.4 Hardness test

Hardness values were recorded from Vickers hardness tester by application of 20Kg load at room temperature. The average of five readings for each type of specimen is reported.

3.1.5 XRD analysis:

X-ray diffraction technique was employed for phase analysis with Philips PANalytica x-ray diffractometer. Test was conducted at 30KV, 20mA, at a scanning rate of 2° per minute for scanning range of 40°-90°. The diffraction patterns were analyzed using Xpert Highscore and JCPDS and the results are shown.

Result and discussion

4.1 Mechanical properties:

UTS, 0.2% YS, percentage elongation and hardness for the test specimens are shown in table 1. It can be seen that for the specimen hold at 700°C (AN 1) the tensile strength & hardness are decreased, whereas ductility is increased marginally (185%) as compared to the as cast and the specimen hold for 90 mins at 1000°C. This phenomenon was observed due to the longer holding period in case of specimen AN 1than specimen AN 2 which was held at 1000°C just for 90 mins. For the specimen which was cooled to room temp. after 90 mins (AN 2), UTS & hardness are found to be less than the as cast specimen but more than the specimen which was hold at 700°C for 330 mins. Percentage elongation for AN 2 has increased to some extent but is very less than AN 1. Yield strength for both AN 1 & AN 2 has increased as compared to the as cast sample but to a higher extent for the latter case.

Table 4: Mechanical Properties of as cast & annealed specimen

SPECIMEN	UTS (MPa)	0.2% YS (MPa)	% El	HARDNESS
AS CAST	522.0	192.57	18.95	217 HV20
AN 1	349.7	199.4	35.16	124 HV20
AN 2	430.4	227.9	19.22	153.2 HV20

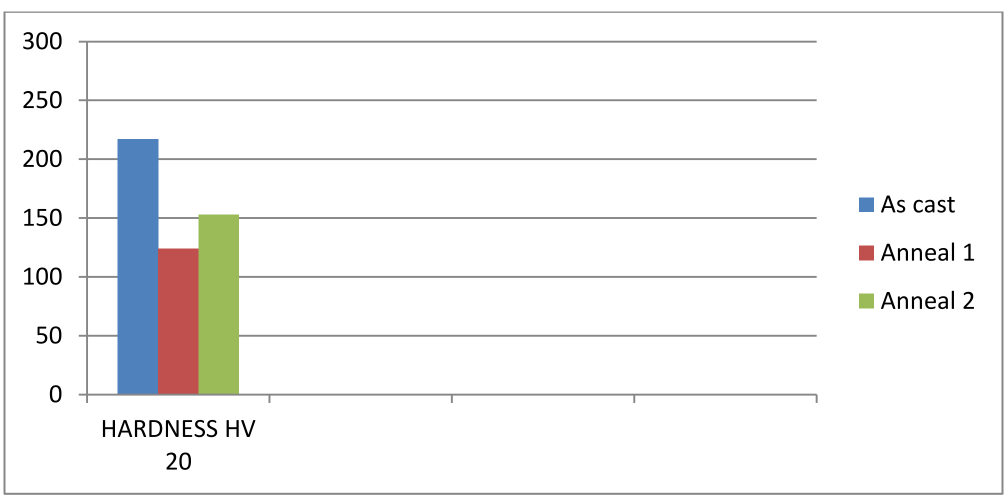


Fig11 : Hardness of as cast and annealed specimen

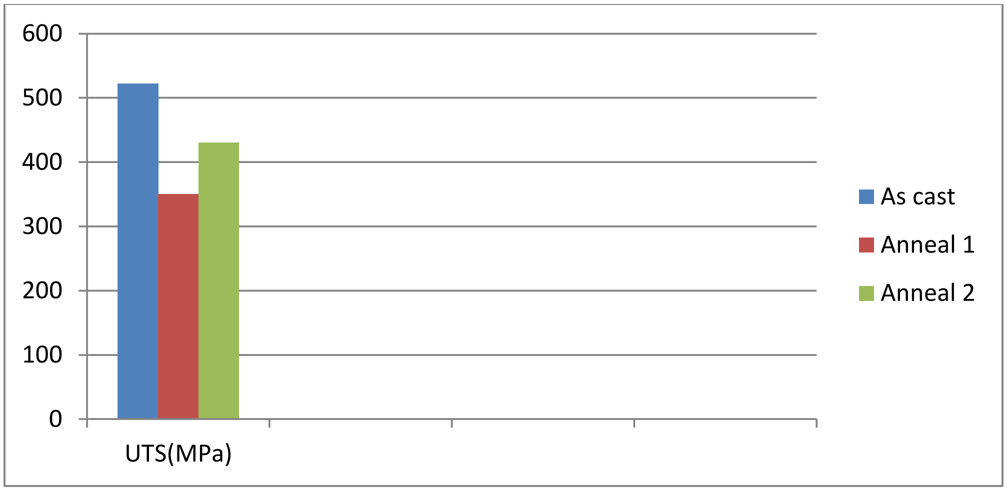


Fig 12 :UTS of as cast and annealed specimen

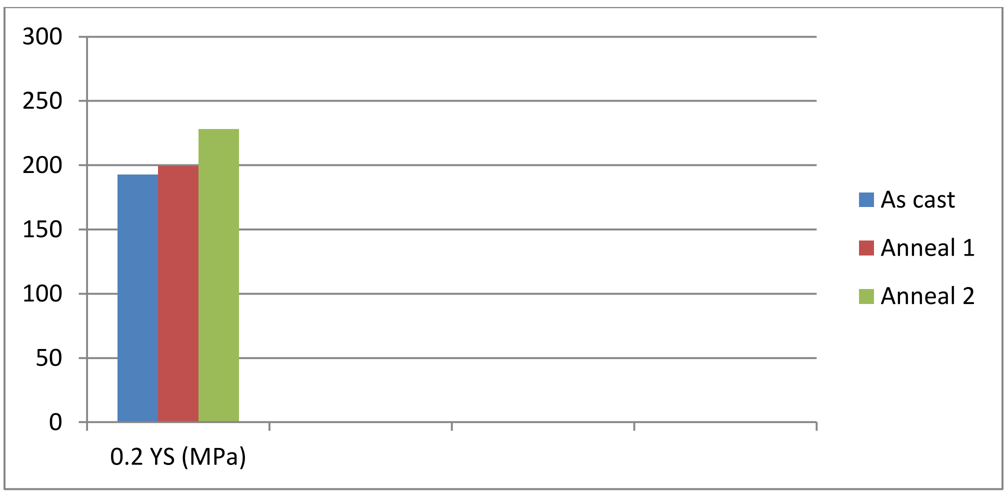


Fig 13 :Yield strength of as cast and annealed specimen

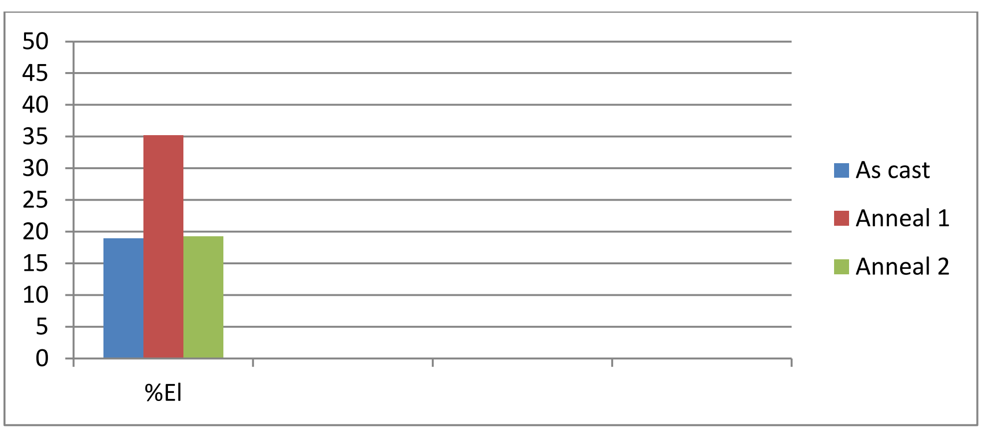


Fig 14 :Elongation of as cast and annealed specimen

4.2 Fractography:

The SEM images for treated and as cast specimens are shown in fig 15. Every specimen when observed under 50X magnification level was found to have the graphite nodules uniformly distributed throughout the ferrite matrix. When closely viewed at 250X magnification the dimple type fracture mechanism was observed in each of the treated sample. However in case of as cast sample the fracture surface was found to be brittle in nature. The reason behind this could be non uniformity in cooling during casting with respect to the specimen geometry which lead to presence of residual stresses in the matrix. On the other hand both the annealed specimens show better homogenization of matrix resulting in ductile fracture. It is clear from the SEM images that for a longer dwelling time the material will be more ductile in nature.

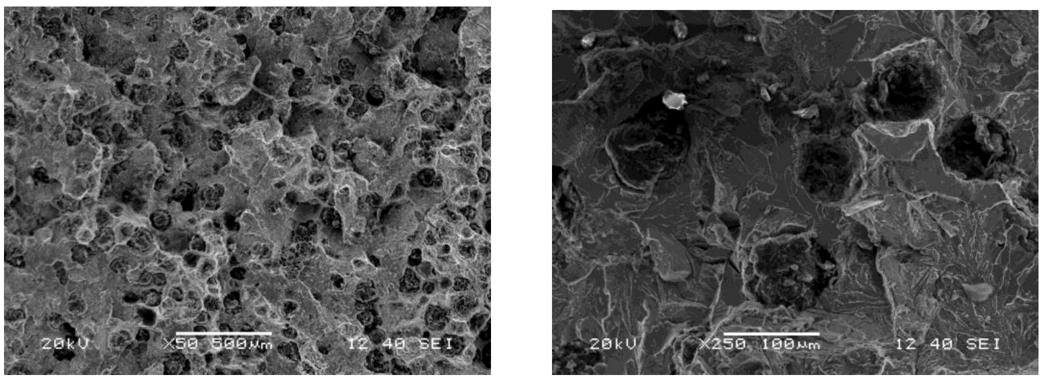


Fig 15. (a): AS-CAST Specimen at 50X & 250X

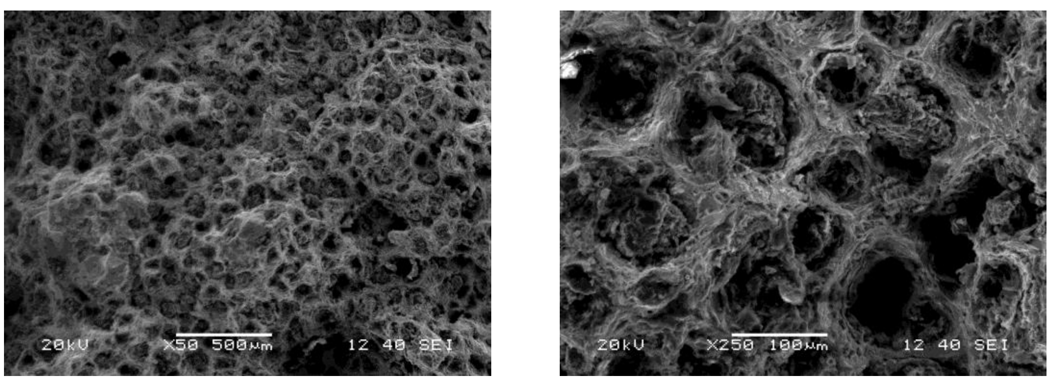
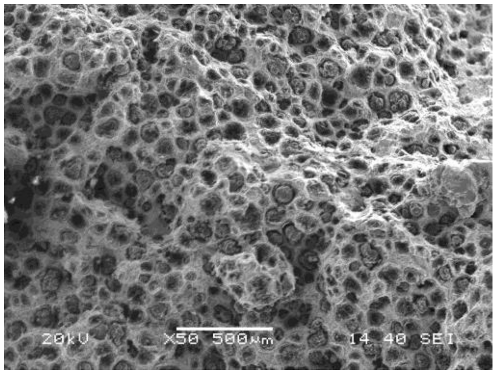


Fig 15. (b): AN 1 Specimen at 50X & 250X



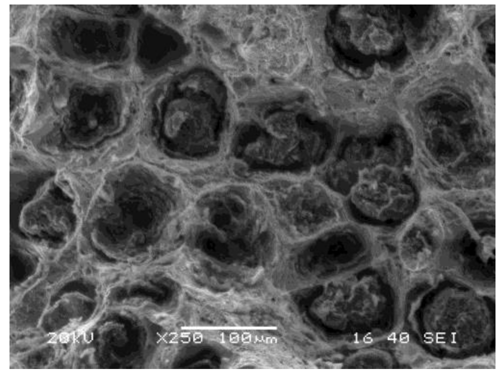
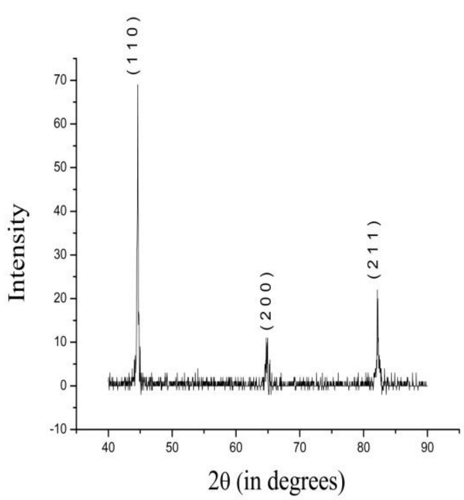


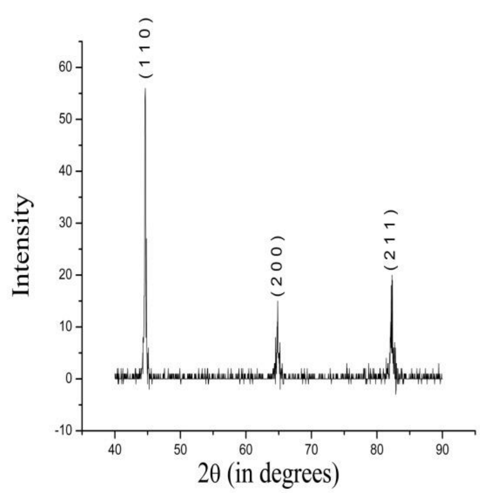
Fig 15 (c): AN 2 Specimen at 50X & 250X

4.3 XRD analysis:

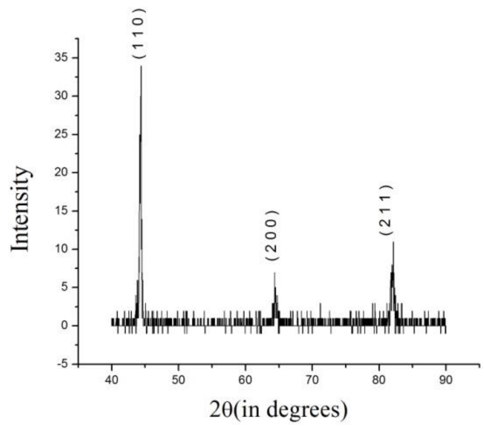
The XRD pattern for each type of specimen is shown in fig 4 below. It can be clearly seen that the peak intensity for (1 1 0) plane of as cast specimen is more than that of AN 1 & AN 2 specimen. This could be the result of change in orientation of the atoms, change in planar density and atomic packing factor. But in case of (2 0 0) plane the intensity of as cast specimen does not vary as much compared to the specimen cooled from 1000°C, is less than the AN 1 specimen. The increase in peak intensity for (2 0 0) plane could be the result of shifting of atoms from the (1 1 0) plane due to the change in orientation. During the analysis of the XRD pattern with JCPDS software ferrite phase was found for each type of specimen



(a)



(b)



(c)

Fig 16. XRD Pattern; (a) AS-CAST Specimen, (b) AN 1 Specimen, (c) AN 2 Specimen

CONCLUSION

1. . When closely viewed at 250X magnification the dimple type fracture mechanism was observed in each of the treated sample. However in case of as cast sample the fracture surface was found to be brittle in nature. The reason behind this could be non-uniformity in cooling during casting with respect to the specimen geometry which lead to presence of residual stresses in the matrix. On the other hand both the annealed specimens show better homogenization of matrix resulting in ductile fracture.

2. It is found that peak intensity for (1 1 0) plane of as cast specimen is more than that of AN 1 & AN 2 specimen. This could be the result of change in orientation of the atoms, change in planar density and atomic packing factor.

3. In case of (2 0 0) plane the intensity of as cast specimen does not vary as much compared to the specimen cooled from 1000°C, is less than the AN 1 specimen. The increase in peak intensity for (2 0 0) plane could be the result of shifting of atoms from the (1 1 0) plane due to the change in orientation.

4.On observing the hardness value , yield strength and ductility it is seen that anneal 1 has the lowest hardness and yield strength but maximum ductility .This is due to the formation of more ferrite phase in anneal 1 sample due to slower cooling rates and homogenization due to isothermal holding.

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